

PATENT SPECIFICATION

1041020



NO DRAWINGS

1041020

Date of Application and filing Complete Specification: Aug. 28, 1963.

No. 34117/63.

Application made In United States of America (No. 270,808) on April 5, 1963.

Complete Specification Published: Sept. 1, 1966.

© Crown Copyright 1966.

Index at acceptance:—D1 B(2K1B, 2K1C1, 2K1D, 2K1E, 2K3A, 2K3B1, 2K3B4, 2K3B7); B2 E1H

Int. Cl.:—D 06 p

COMPLETE SPECIFICATION

Dyeing and or Printing of Cellulose Acetate

We, UNITED MERCHANTS AND MANUFACTURERS, INC., a corporation organised and existing under the laws of the State of Delaware, United States of America, of 1407 Broadway, New York 18, New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to processes of dyeing and/or printing cellulose acetate yarns and fabrics and, more particularly, to the pretreatment of cellulose acetate yarns and fabrics to condition them so that they readily and uniformly receive dyes, especially dyes for which they normally have little substantivity and/or affinity.

In this specification all percentage values are given on a weight basis.

Textiles—namely, yarns and fabrics composed wholly or partially of cellulose acetate fibers or filaments—cannot by presently known techniques be colored—i.e., dyed or printed—employing many of the available dyes such as the reactive dyes. In many cases, it is impossible to attain an identical hue on yarns and fabrics containing cellulose acetate fibers along with other fibers—for example, cotton fibers. Nor is it possible to dye or print on cellulose acetate yarns and fabrics employing, for example, the reactive dyes commercially available which dyes are now popular because of the bright colors, fast to washing and sunlight, produced thereby on fibers, chiefly cellulosic, which have a marked affinity therefor. For example, attempts to color a mixture of cotton and cellulose acetate fibers a bright turquoise shade by heretofore known techniques result in failure because the acetate portion of the yarn or fabric will always be duller than the cotton. There are no bright turquoise dyestuffs in the dispersed dyestuff range which

has to be used to color the cellulose acetate and hence it is not feasible with presently available techniques to produce bright uniform turquoise shades on blends of cotton and cellulose acetate. The difficulty of uniformly coloring such blends is also compounded by the further difficulty of obtaining equal fastness properties with the two ranges of dyestuffs which must be used. The fastness properties of the dyestuffs on each fiber can differ widely with respect to resistance to exposure to such influences as light, water and solvents. Each fiber can also require a different method for the final fixation of the dyestuff, hence the processing of yarns and fabrics composed of blends of fibers by heretofore known techniques is invariably much more complex than it is for yarns and fabrics composed of fibers all of the same chemical composition.

One method which has been proposed as a solution for these difficulties is the coloration of such fabrics and yarns by applying thereto either an oil-in-water or water-in-oil pigmented emulsion. While this method does produce uniformly colored yarns and fabrics it leaves much to be desired. The resulting yarn or fabric is usually stiffened considerably and if the blended yarn or fabric has a high proportion of synthetic fibers it usually has poor fastness to dry cleaning, solvent spotting and rubbing. The range of hues producible by such pigmented printing and dyeing techniques also leaves much to be desired and bright hues, as a general rule, cannot be produced by presently available emulsions.

By cellulose acetate, as used herein, is meant cellulose diacetate (Celanese) and cellulose triacetate (Arnel and Tricel). The words "Celanese," "Arnel" and "Tricel" are Trade Marks.

It is the principal object of this invention to provide a process for conditioning the surface of yarns and fabrics composed of all cellulose acetate or containing at least 10%

[Price 4s. 6d.]

45
50
55
60
65
70
75
80
85

BEST AVAILABLE COPY

of cellulose acetate, so that they can be uniformly and readily dyed and/or printed to produce pleasing effects including bright hues heretofore not obtainable, and particularly can 5 be colored with dyestuffs for which cellulose acetate normally has little or no affinity.

Other objects and advantages of this invention will be apparent from the following detailed description thereof.

10 In accordance with this invention the cellulose acetate containing yarns or fabrics including such yarns or fabrics composed wholly of cellulose acetate, are treated before dyeing or printing with an aqueous alkali solution of hydroxyethylcellulose and a coating then produced by coagulation to form a water-insoluble hydroxyethylcellulose firmly bonded to the cellulose acetate fibers, which water-insoluble hydroxyethylcellulose has surprisingly good 15 substantivity and/or affinity for readily available dyestuffs such as the reactive dyes, vat dyes, azoic dyes, naphthol shades, chrome dyes, direct dyes and sulfur dyes, applied by conventional dyeing techniques. Cellulose acetate 20 blends containing fibers of markedly different dyeing characteristics when treated by the process of this invention are dyed or printed with uniform coloration in bright colors, and the dyeings or prints possess good fastness properties to light, heat, water, solvents and abrasion.

25

The coagulated and regenerated hydroxyethylcellulose provides a cellulosic surface over the cellulose acetate yarn or fabric which is receptive to dyes used in dyeing or printing cellulosic materials. The hydroxyethylcellulose used is preferably produced by reacting alkali cellulose with ethylene oxide so that from 2% to 35 10%, advantageously from 2% to 5%, of ethylene oxide is actually combined with the alkali cellulose. The preferred hydroxyethylcellulose is that sold commercially under the trademark "Ethylose" which has an ethylene oxide content of about 4.0% and has the 40 hydroxy groups evenly distributed along the cellulose chain and evenly substituted with ethyl substituents, the degree of substitution being about 0.15. Films produced as herein described are remarkably resistant to swelling 45 in water, have good affinity for dyestuffs and result in colored products which are fast to washing, sun, abrasion and dry cleaning.

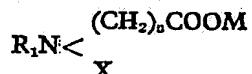
50 The concentration of hydroxyethylcellulose in the solution applied to the cellulose acetate textile are preferably varied from 2% to 8%. A more preferred concentration for this solution is about 5%. The amount of alkali metal hydroxide, e.g., sodium or potassium hydroxide, in this solution to obtain proper solubility of the hydroxyethylcellulose is from 5% to 60 9%, preferably about 6.25%. Preferably the solution also contains a small amount of an amphoteric detergent, desirably from 0.1% to 1%. Thus the hydroxyethylcellulose solution 65 preferably contains from 2% to 8% hydr-

oxyethylcellulose, from 5% to 9% alkali metal hydroxide, from 0.1% to 1%, amphoteric detergent, and the rest water. A preferred solution contains:

| | | |
|-----------------------|-------|----|
| Hydroxyethylcellulose | 5.00% | 70 |
| Sodium Hydroxide | 6.25% | |
| Amphoteric Detergent | 0.25% | |
| Water | 88.5% | |
| Total | 100% | |

As the amphoteric detergent, the alkali metal, e.g., sodium or potassium salt of an imidazoline C₁₀ to C₁₄ aliphatic carboxylic acid is preferred. This amphoteric surfactant is available commercially under the trade name "Uniterge K" from the Universal Chemical Corporation of Central Falls, Rhode Island, United States of America. It has the surprising property of promoting the penetration and distribution of the hydroxyethylcellulose into and around the fibers and/or films of cellulose diacetate and cellulose triacetate. Other amphoteric detergents which can be used are:

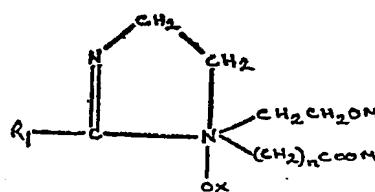
(A) Deriphat compounds (trademark of General Mills, United States of America), such as sodium N-lauryl β-aminopropionate; these compounds have the formula:



in which R₁ is an alkyl group containing from 8 to 19 carbon atoms; n is 1 or 2; M is sodium, potassium, ammonium, monoethanolamine, diethanolamine or triethanolamine; and X is hydrogen or an ethoxylated group having from 1 to 20 carbon atoms.

(B) The alkali metal salts of the ethoxylated acyl alkyl amines, in which the ethoxylated groups have from 1 to 20 carbon atoms, preferably 10 to 15, exemplified by Triton QS-15 (the word "Triton" is the trademark of Rohm & Haas Co., United States of America).

(C) Miranol Compounds (trademarks of Miranol Chemical Company, United States of America), which are compounds having the formula:



in which R₁ is an alkyl group having from 8 to 19 carbon atoms; n is 1 or 2; M is sodium, potassium, ammonium, monoethanolamine, diethanolamine or triethanolamine; and X is

| | | |
|---|---|-----|
| hydrogen or an ethoxylated group having from 1 to 20 carbon atoms. | be accomplished through the removal of the alkali metal hydroxide either through neutralization, dehydration, gelation through heating, followed by washing, or a combination of any of the foregoing. Coagulants and regenerants which can be used are as follows: | 70 |
| The solution is prepared at temperatures below 77° F. A portion of the water can be added as ice to facilitate production of the solution. Lower temperatures facilitate dissolution of the hydroxyethylcellulose. At temperatures above 77° F. there is a tendency for gelation to take place. The amphoteric detergent is dissolved in water while agitating. The amphoteric nature of this surfactant insures good stability in the alkaline medium necessary to dissolve the hydroxyethylcellulose and provides for a good wetting action of the hydroxyethylcellulose on the textile material. It also facilitates the dispersion of the hydroxyethylcellulose when added to the water containing the amphoteric detergent. After a good dispersion is obtained the sodium hydroxide is added followed by the addition of about one-fifth the water content as ice. The use of ice accelerates solution and also improves the filterability of the solution. After filtration the solution can be used but it is preferred to allow it to age for about 24 hours. Such aging lowers the viscosity of the solution thereby making it easier to apply to the textile material. | A) Alkaline Coagulants (1) Sodium Hydroxide solution of mercerizing strength, i.e., 20% to 30% (2) Solutions of Sodium Carbonate (3) " " Sodium Bicarbonate (4) " " Borax (5) " " Trisodium Phosphate (6) " " Disodium Phosphate (7) " " Sodium Silicate | 75 |
| The hydroxyethylcellulose solution thus prepared can be applied in any conventional manner such as by spraying, coating or padding. The amount of hydroxyethylcellulose solution deposited on the textile can vary between 10% to 100% of the textile material's weight. A preferred amount is approximately 80% based on the dry weight of the textile. | B) Acid Coagulants (1) Solutions of water soluble acids, such as a) Sulfuric Acid b) Phosphoric Acid c) Acetic Acid d) Formic Acid (2) Solutions of acid salts or salts and acid, such as a) Sodium Sulfate and Sulfuric Acid b) Sodium Phosphate and Phosphoric Acid c) Aluminum Sulfate and Sulfuric Acid (3) Vapors of various acids, such as a) Carbon Dioxide Gas b) Acetic Acid Vapor c) Formic Acid Vapor | 80 |
| After the application of the hydroxyethylcellulose solution to the cellulose acetate yarns or fabrics, the latter is treated to convert the hydroxyethylcellulose to a water-insoluble form. Preferably, but not necessarily, the regeneration is preceded by a steaming treatment. Such steaming is effected by introducing the textile material impregnated with the hydroxyethylcellulose into a steaming chamber maintained at a temperature of 200° F. to 250° F. and exposing it to this atmosphere for 10 seconds to 50 seconds. Preferred steaming conditions are about 240° F. with a dwell time of 30 seconds. This steaming causes the hydroxyethylcellulose solution to more thoroughly penetrate into the cellulose acetate. At the steaming temperatures, the viscosity of the hydroxyethylcellulose decreases by approximately 2000 to 3000 centipoises, probably followed by some gelation of the hydroxyethylcellulose. Such gelation at this stage is not detrimental but is advantageous in preventing loss of hydroxyethylcellulose during the subsequent coagulating and regenerating treatment. | Treatment with acid media effects neutralization of the alkali metal hydroxide with consequent precipitation of the hydroxyethylcellulose in water-insoluble form. | 90 |
| The deposited hydroxyethylcellulose, whether or not subjected to a steaming treatment, can be coagulated and regenerated by a number of methods. The regeneration can | C) Neutral Coagulants (1) Solutions of Ammonium Salts a) Ammonium Sulfate Solution b) Ammonium Sulfate and Sodium Sulfate Solution D) Heat (1) Hot Air (2) Infra Red (3) Steam | 100 |
| | Except for coagulating and regeneration procedures described in class D, all of the procedures are best conducted at temperatures of from 40° C. to 60° C. Class D procedures are carried out at any convenient temperature controlled by the heating medium used. In all cases the coagulation and regeneration procedure effects precipitation of the hydroxyethylcellulose in a form which bonds with the fibers of the textile material. | 105 |
| | Coagulation and regeneration should in all cases be followed by rinsing with water or by neutralizing or acidifying, and rinsing to remove by-products such as salts and excess acid or alkaline materials. | 115 |
| | | 120 |
| | | 125 |
| | | 130 |

A preferred procedure is the use of a solution of ammonium sulfate and sodium sulfate at a temperature of 50° C. Equally good results can be obtained using a solution of 5 10% sulfuric acid and 15% sodium sulfate at 50° C. Such solutions produce hydroxyethylcellulose films on the fibers of the cellulose acetate textile materials with the least amount of stiffening and with a minimum 10 tendency to reswell upon subsequent exposure to water.

The thus treated cellulose acetate textile

materials can be dyed with commercially available dyestuffs used for cellulosic materials, employing conventional dyeing and/or printing techniques used with such dyestuffs and with the production of uniform colorations fast to washing, dry cleaning, solvent spotting and rubbing on the textile materials, including yarns and fabrics composed of blends of different fibers having markedly different dye affinities. Typical suitable dyestuffs are listed below:

15

20

| A | REACTIVE DYES: | PART I C.I. |
|---|------------------------------|-------------------------|
| | Procion Yellow 6 G | C.I. Reactive Yellow I |
| | Cibacron Brilliant Yellow 3G | C.I. Reactive Yellow 2 |
| | Levafix Yellow 4 G | C.I. Reactive Yellow 10 |
| | Drimarene Yellow Z—3 GL | C.I. Reactive Yellow 11 |
| | Remazol Golden Yellow Y | No Color Index No. |
| | Procion Brilliant Orange G | C.I. Reactive Orange 1 |
| | Cibacron Brilliant Orange G | C.I. Reactive Orange 2 |
| | Drimarene Orange 2—G | C.I. Reactive Orange 6 |
| | Remazol Brilliant Orange RR | No Color Index No. |
| | Procion Brilliant Red 2 B | C.I. Reactive Red 1 |
| | Cibacron Brilliant Red 3 B | C.I. Reactive Red 4 |
| | Reactone Red 2 B | C.I. Reactive Red 17 |
| | Remazol Brilliant Red BB | No Color Index No. |
| | Procion Purple H 3 RS | C.I. Reactive Violet I |
| | Cibacron Violet 2 R | C.I. Reactive Violet 2 |
| | Remazol Red Violet R | No Color Index No. |
| | Drimarene Violet Z—RL | C.I. Reactive Violet 3 |
| | Procion Blue 3 G | C.I. Reactive Blue 1 |
| | Procion Brilliant H 7 G | C.I. Reactive Blue 3 |
| | Cibacron Brilliant Blue BR | C.I. Reactive Blue 5 |
| | Drimarene Blue ZGL | C.I. Reactive Blue 16 |
| | Remazol Brilliant Blue R | No Color Index No. |
| | Levafix Brilliant Blue RR | C.I. Reactive Blue 12 |
| | Levafix Brilliant Green IB | C.I. Reactive Green I |
| | Procion Brilliant Green 2 B | No Color Index No. |
| | Cibacron Brown 3 GR | C.I. Reactive Brown I |
| | Procion Brown H 4R | No Color Index No. |
| | Procion Black HN | No Color Index No. |
| | Cibacron Black BG | C.I. Reactive Black I |
| | Remazol Black B | No Color Index No. |
| | Drimarene Black Z BL | C.I. Reactive Black 4 |
| | Levafix Grey IG | C.I. Reactive Black 2 |

The words "Procion" and Cibacron are Trade Marks.

| B | VAT DYES | PART I | PART II |
|---|-------------------------------|---------------------|-------------------------------------|
| | Ponsol Flavone GC | C.I. Vat Yellow 2 | 67300 |
| | Algol Orange RFN | C.I. Vat Orange 5 | 73335 |
| | Ciba Brilliant Pink R | C.I. Vat Red I | 73360 |
| | Indanthrene Scarlet 2G | C.I. Vat Red 14 | 71110 |
| | Sandothrene Violet 4NR | C.I. Vat Violet I | 60010 |
| | Ciba Blue 2 B | C.I. Vat Blue 5 | 73065 |
| | Indanthrene Brilliant Green B | C.I. Vat Green I | 59825 |
| C | AZOIC DYES | PART I | PART II |
| | Rapidogen Yellow G | C.I. Azoic Yellow I | 37610/37090 |
| | Rapidogen Orange RFN | C.I. Azoic Orange 3 | 37558/37010 |
| | Rapidogen Red G | C.I. Azoic Red 6 | 37520/37090 |
| | Rapidogen Violet B | C.I. Azoic Violet I | 37505/37165 |
| | Rapidogen Blue B | C.I. Azoic Blue 6 | 37505/37175 |
| | Rapidogen Green B | C.I. Azoic Green I | 37585/37175 |
| | Rapidogen Black Brown T | C.I. Azoic Brown 16 | 37605/37120 |
| | Rapidogen Black 3G | C.I. Azoic Black 4 | NONE |
| D | NAPHTHOL SHADES | | |
| | Naphthol AS—RL | Part I Part II | New C.I. 11; New C.I. 37535, and |
| | Fast Red B Salt | Part I Part II | New C.I. 5; New C.I. 37125 |
| | Naphthol AS—ITR | Part I Part II | New C.I. 12; New C.I. 37550, and |
| | Fast Red ITR Salt | Part I Part II | New C.I. 42; New C.I. 37150 |
| | Naphthol AS—L3G | Part I Part II | New C.I. 33; New C.I. 37620, and |
| | Fast Red AL Salt | Part I Part II | New C.I. 36; New C.I. 37275 |
| | Naphthol HB | Part I Part II | New C.I. 16; New C.I. 37605, and |
| | Fast Orange GC Salt | Part I Part II | New C.I. 2; New C.I. 37005 |
| | Naphthol AS—RL | | C.I. given above, and |
| | Fast Red RL Salt | Part I Part II | New C.I. 34; New C.I. 37100 |

D NAPHTHOL SHADES (Cont.)

| | | |
|----------------------|---------|--|
| Naphthol AS—PH | Part I | New C.I. 14; |
| Fast Red AL Salt | Part II | New C.I. 37558, and C.I. given above |
| Naphthol AS—PH | | C.I. given above, and |
| Fast Orange RD Salt | Part I | New C.I. 49 |
| Naphthol AS—BS | Part I | New C.I. 17; |
| Fast Scarlet RN Salt | Part II | New C.I. 37515, and Part I New C.I. 13; Part II New C.I. 37130 |

E CHROME DYES

| | PART I | PART II |
|------------------------------|------------------------|---------|
| (1) Chrome Luxine Yellow 5G | NONE | NONE |
| (2) Chromocitronine R | C.I. Mordant Yellow 26 | 22880 |
| (3) Chrome Fast Orange RL | C.I. Mordant Orange 37 | 18730 |
| (4) Chrome Fast Red NL | NONE | NONE |
| (5) Printing Chrome Brown DS | NONE | NONE |
| (6) Panduran Blue B | NONE | NONE |
| (7) Panduran Turquoise | NONE | NONE |
| (8) Panduran Green G | NONE | NONE |
| (9) Chrome Luxine Green S | Mordant Green 3. | NONE |
| (10) Novochrome Fast Grey N | NONE | NONE |

F DIRECT DYES

| | PART I | PART II |
|--------------------------|-----------------------|---------|
| Fastusol Orange LS GL—CF | C.I. Direct Orange 59 | NONE |
| Fastusol Red L4 BL—CF | C.I. Direct Red 79 | 29065 |
| Pontamine Fast Blue 4 GL | C.I. Direct Blue 78 | 34200 |

G SULFUR DYES

| | PART I | PART II |
|-----------------------------|-------------------------------------|---------|
| Sodyesul Liquid Yellow E—CF | C.I. Solubilized Sulfur Yellow 4 | 53125 |
| Sodyesul Liquid Green BG—CF | C.I. Solubilized Sulfur Green 16 | NONE |
| Sodyesul Liquid Black 4G—CF | C.I. Solubilized Sulfur Black 1 | 53185 |

In the examples given below the cellulose acetate was first conditioned by the following typical four-step procedure:

5 Step (1) The cellulose acetate fabric is impregnated with a solution of 5% hydroxyethylcellulose, .25% "Unterge K," 6.25% sodium hydroxide and 88.50% water. This impregnating step can be conducted in a conventional two bowl textile padder, at room temperature. The expression of the padder is set so that the textile material absorbs approximately 80% of its dry weight of the impregnating solution.

10 Step (2) This optional, but preferred, step consists of steaming the textile material from Step (1) in a continuous horizontal steamer at 250°F. for 30 seconds.

15 Step (3) In this step the regeneration and coagulation of the hydroxyethylcellulose film is conducted in a conventional 8-compartment open washer. Each compartment is filled with solutions in accordance with the descriptions which follow:

20 Compartment 1: 15% Ammonium Sulfate
5% Sodium Sulfate
80% Water
Temperature 122° F.

25 Compartment 2: Overflowing cold water.
Compartment 3: 5% Sulfuric Acid conc.
10% Sodium Sulfate
85% Water
Temperature 122° F.

30 This provides a neutral bath.
Compartment 4: Overflowing cold water.
Compartment 5: 0.25% Sodium Carbonate
Temperature 180° F.

35 Compartment 6: 0.25% Sodium Carbonate
Temperature 180° F.

40 In this compartment neutralization of the caustic soda employed in Step (1) is effected to convert the hydroxyethylcellulose to an insoluble form.

45 Compartment 7: Overflowing cold water.

50 Compartment 8: Hot Water.
Temperature 180° F.

55 In Compartments 5 and 6 excess acid used in Compartment 3 is neutralized.

60 Compartment 7: Overflowing cold water.
Compartment 8: Hot Water.
Temperature 180° F.

Step (4) After completion of Step (3) the textile material has a pH of about 7 and is dried in any conventional manner such as through the use of drying cylinders, an enclosed tenter frame or a loop dryer. 65

The textile material is now ready for printing or dyeing with dyestuffs normally used for cellulose, such as those above disclosed, by use of any conventional application techniques. 70

The following examples are given to illustrate this invention. It will be appreciated that this invention is not limited to these examples. 75

EXAMPLE I

A textile material consisting of 50% cellulose diacetate (Celanese) and 50% cotton was pretreated according to the methods of this invention as has been previously described. 80 It was then printed by conventional methods using a roller printing machine and an intaglio engraved roller with a paste of the following composition:

| | |
|--|----|
| 5. % Procion Brilliant Blue H7GS | 85 |
| 15. % Urea | |
| 35. % Sodium Alginate Thickening | |
| 1. % Sodium Meta Nitro Benzene Sulfonate | |
| 1.5% Sodium Bicarbonate | 90 |
| 42.5% Water | |

After printing the textile material was steamed for five minutes, rinsed, soaped and dried. A uniform turquoise blue print was obtained on both the cotton and cellulose acetate portions of the textile material. This shade was fast to washing and was commercially acceptable. A comparison print made on the untreated textile material was much inferior in appearance, and the cellulose acetate portion was completely white. The procion dye used in the print paste is a derivative of cyanuric chloride, manufactured by Imperial Chemical Industries Ltd., and requires the presence of hydroxyl groups on the fiber for fixation of the dye to occur. 100

EXAMPLE Ia

This Example was a substantial duplication of Example I except that it involved the treatment of a textile material consisting of 50% of cellulose triacetate (Arnel) and 50% cotton. 110

EXAMPLE II

The textile material used in Example I was dyed by impregnating on a two bowl padder with the following solution containing a 115 Cibacron dye and possessing one reactive group:

2. % Cibacron Brilliant Orange G
 10. % Urea
 10. % Sodium Alginate Thickening
 1. % Sodium Meta Nitro Benzene
 5 Sulfonate
 1.5% Sodium Carbonate
 75.5% Water

After impregnation the textile material was dried and fixation of the dye was carried out 10 by heat treating, in a conventional textile curing oven, for three minutes at a temperature of 230° F. The material was then washed and dried. The textile material thus processed was dyed a bright uniform orange shade. A textile 15 material not pretreated, on the other hand, when dyed in the same manner had only the cotton fibers dyed with the cellulose acetate fibers substantially free of color.

EXAMPLE III

20 4% Remazol Red Violet R was printed as in Example I on the same textile material. After fixation by steaming and following rinsing, washing and drying, an examination indicated that the fabric was uniformly 25 colored in the printed areas.

EXAMPLE IV

A fabric consisting of 100% cellulose triacetate (Arnel) was treated as hereinabove described to produce thereon a regenerated 30 and coagulated film of hydroxyethylcellulose. It was then dyed by impregnating it on a conventional textile padder with a solution of the following composition:

2. % Procion Brilliant Orange G
 35 20. % Urea
 5. % Keltex (3% Solution of Sodium Alginate)
 1. % Ludigol (Sodium Meta Nitrobenzene Sulfonate)
 40 1.5% Sodium Bicarbonate
 70.5% Water

The fabric was then dried and the fixation 45 of the dyestuff was carried out by heating the fabric at 250° F. for 3 minutes. The fabric was then rinsed and soaped to remove any unfixed dyestuff.

50 A uniform orange shade resulted. For comparative purposes an untreated piece of cellulose triacetate (Arnel) was also dyed in the same manner. The untreated cellulose acetate fabric was practically colorless.

EXAMPLE IVa

This Example was substantially the same as Example IV except for the substitution 55 of a fabric consisting of 100% cellulose diacetate for the cellulose triacetate used in Example IV. The results were the same, namely, the cellulose diacetate was dyed a uniform orange shade.

60 It will be noted that the newer fiber-reactive dyestuffs derived, for example, from cyanuric chloride, vinyl sulphone and tri and

tetrachloro pyrimidine can be used with excellent results in dyeing and/or printing cellulose acetate yarns and fabrics treated in accordance with this invention. These dyes are not suitable for application to cellulose acetate yarns and fabrics not pretreated as herein disclosed. These dyestuffs require the presence of hydroxyl groups within the fiber for fixation of the dyestuff to occur through the formation of a covalent bond. Unless the fiber/dyestuff reaction occurs, these dyes will have no acceptable fastness properties and will be easily removed from the fiber during washing, as invariably carried out following the dyeing or printing of the textiles. The fixation of these dyestuffs on fibers, containing no hydroxyl groups, or on fibers containing relatively few hydroxyl groups, which are not capable of reaction because of inaccessibility through steric hindrance, is of great commercial importance. Through the use of these dyestuffs on cellulose acetate textile materials treated in accordance with this invention, it is possible to obtain shades on cellulose acetate containing fabrics which heretofore had not been attainable.

The fabrics and yarns thus produced which in the case of blends are composed of cellulosic fibers blended with cellulose acetate, including cellulose diacetate, cellulose triacetate and saponified diacetate have a continuous film of water-insoluble hydroxyethylcellulose bonded thereto. The amount of hydroxyethylcellulose film on the material is from 4% to 7%, preferably about 5%, based on the dry weight of the cellulose acetate yarns or fabrics heated.

WHAT WE CLAIM IS:—

1. The process of conditioning cellulose acetate yarns and fabrics to render them receptive to dyes having affinities for cellulosic materials, which process comprises applying to the cellulose acetate yarns and fabrics an alkaline solution of hydroxyethylcellulose, and thereafter coagulating and regenerating the hydroxyethylcellulose to convert it to a water-insoluble film bonded to the yarns and fabrics.

2. The process as defined in claim 1 in which an alkaline solution of hydroxyethylcellulose containing an amphoteric detergent is applied to the cellulose acetate yarns and fabrics, and thereafter the hydroxyethylcellulose is coagulated and regenerated to convert it to a water-insoluble film bonded to the yarns and fabrics.

3. The process as defined in claim 2 in which the alkaline solution of hydroxyethylcellulose contains from 2% to 8% by weight of hydroxyethylcellulose, from 5% to 9% by weight of alkali metal hydroxide, and from 0.1% to 1% of an amphoteric detergent in an amount to apply to the cellulose acetate yarns and fabrics from 10% to 100% of

65

70

75

80

85

90

95

100

105

110

115

120

125

the hydroxyethylcellulose solution.

4. The process as defined in claim 3 in which the yarns and fabrics treated with said alkaline solution of hydroxyethylcellulose is steamed at a temperature from 200° F. to 250° F. for from 10 seconds to 50 seconds prior to coagulating and regenerating the hydroxyethylcellulose.

5. The process as defined in claim 2 in which the amphoteric detergent is the alkali metal salt of an imidazoline C_{10} to C_{18} aliphatic carboxylic acid.

6. The process as defined in claim 1 in which the coagulation and regeneration of the hydroxyethylcellulose is effected by treatment with an alkaline solution of sufficient strength to precipitate the hydroxyethylcellulose as a water-insoluble film on the cellulose acetate yarns and fabrics, and subsequent removal of the alkali.

7. The process as defined in claim 1 in which the coagulation and regeneration of the hydroxyethylcellulose is effected by treatment of the yarns and fabrics containing an alkaline solution of hydroxyethylcellulose with an acid to neutralize the alkalinity of the hydroxyethylcellulose solution on the cellulose acetate yarns and fabrics and effect the precipitation of the hydroxyethylcellulose in a water-insoluble form to produce the said film of hydroxyethylcellulose on the cellulose acetate yarns and fabrics.

8. The process as defined in claim 1 in which the coagulation and regeneration of the hydroxyethylcellulose applied to the cellulose acetate yarns and fabrics is effected by heating the said yarns and fabrics to effect precipitation of the hydroxyethylcellulose in a water-insoluble form, and subsequently re-

moving the alkali and thereby producing the film of hydroxyethylcellulose bonded to the cellulose acetate yarns and fabrics.

40
45
50
55
60
65
70

9. The process as defined in claim 1 in which the hydroxyethylcellulose is produced by reacting an alkali cellulose with ethylene oxide so that 2% to 5%, by weight of ethylene oxide is combined with the alkali cellulose, and applying such solution of hydroxyethylcellulose containing about 5% hydroxyethylcellulose, about 6.25% sodium hydroxide, about 0.25% of a sodium salt of an imidazoline C_{10} to C_{18} aliphatic carboxylic acid in an amount to deposit on the cellulose acetate yarns and fabrics about 80% by weight of said solution based on the weight of said yarns and fabrics.

10. A cellulose acetate yarn or fabric, having bonded to its surface a film of water-insoluble hydroxyethylcellulose, which film has an affinity for dyestuffs for dyeing cellulosic materials.

60
65
70

11. The material defined in claim 10 in which the amount of hydroxyethylcellulose film on said material is from 4% to 7% based on the weight of said material.

12. The novel process herein disclosed of conditioning cellulose acetate yarns and fabrics to impart thereto an affinity for dyes which have little or no affinity for cellulose acetate but have marked affinity for cellulosic materials.

13. The novel conditioned cellulose acetate yarns and fabrics herein disclosed.

REDDIE & GROSE,
Agents for the Applicants,
6, Bream's Buildings,
London, E.C.4.

Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press (Leamington) Ltd.—1966. Published by The Patent Office, 25 Southampton Buildings, London, W.C.2, from which copies may be obtained.

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- BLACK BORDERS**
- IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- FADED TEXT OR DRAWING**
- BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- SKEWED/SLANTED IMAGES**
- COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- GRAY SCALE DOCUMENTS**
- LINES OR MARKS ON ORIGINAL DOCUMENT**
- REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- OTHER:** _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.